



Fabrication and properties of thermal sprayed stainless steel-based nanocomposite coatings



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ABSTRACT

Stainless steel-based feedstock powders with nano-iron oxide particles (from direct addition and thermal oxidation) were prepared by ball milling and oxidation process, respectively. It was found that iron oxide nanoparticles were on the surface of stainless steel powders in the form of particulates (deliberate addition of magnetite and hematite) or nanoplatelets (thermal oxidation). The powders were thermal sprayed by low velocity oxy-fuel (LVOF) technique. The stainless steel-based coatings had the typical thermal spray microstructure, including splats, oxide layers, unmelted particles and pores. There were no nanoparticles seen in the microstructure due to complete oxide melting at the flame spray temperature and iron oxide in all coatings was in the form of hematite. Considering physical properties, porosity was decreased when there were second phase nanoparticles and thickness was very similar except the coating with hematite addition. Hardness of the coatings was slightly increased when incorporating with iron oxide nanoparticles. Sliding wear rate and friction coefficient of the nanocomposite coatings were lower than that of the pure stainless steel. Iron oxide nanoparticles could improve hardness and sliding wear resistance in the stainless steel-based coatings due to an increasing amount of iron oxide in the coatings.

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1. Introduction

Stainless steel is one of the most common metal alloys used nowadays in various applications, including machinery parts in manufacturing industry. It is also known that wear and corrosion in metal compartments is inevitable after a long service time and therefore maintenance or replacement of the parts is necessary. Thermal spray is a solution to maintain the parts where thin layers of coatings were deposited on the parts by melting the desirable metals or alloys and then accelerating the droplets to form splats upon collision—later, the splats will solidify and the coating is created [1]. The advantage of thermal spray technique is that no replacement of spare parts is required and the maintenance is a lot more convenient with lower cost.

Thermal spray is also utilized in stainless steel materials. Zhao and Lugscheider [2] applied different spraying processes (atmospheric plasma spraying, shrouded plasma spraying and high velocity oxy-fuel (HVOF) technique) to produce 316 L stainless steel coatings and it was found that HVOF was the best practice because this method gave the coatings with low oxidation, high hardness and best corrosion resistance. The recent development also gives rise to a novel high pressure

HVOF that has very high deposition efficiency (up to 90%) [3]. On the other hand, the properties of the thermal sprayed coatings can also be improved not only by spraying process but also by feedstock powder preparation process. The method to enhance the mechanical properties of the stainless steel coatings is to incorporate with oxide such as aluminium oxide and zirconium oxide [4,5] or carbide such as titanium carbide [6] to form alloys with high hardness. This is the concept of composite powders where a second phase is introduced to the main phase (stainless steel in this case). Not only the mechanical properties are optimized, flowability of feedstock powder during thermal spray or physical properties of the final coatings (for example, porosity) can also be improved.

It is well known that metal oxides are added to improve mechanical properties of metal-based coatings as second phase particles, which can be incorporated into the main phase by deliberate addition or as products of chemical reaction. In case of metal oxide second phase, oxidation reaction is one possible method to create the second phase particles. For stainless steel based powders, oxidation at high temperatures results in nanostructured metal oxides, which could be iron oxide or oxides of its component (i.e. chromium and nickel). There has been an attempt to produce metal oxide particles from bulk metal sample. Fu et al. [7] first prepared Fe₂O₃ nanowires from oxidation reaction and the wires had the diameter of ~15–75 nm and the length of ~10–20 μm. Thermal

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oxidation of Fe foil in air was carried out by Grigorescu et al. [8] and it was found that the best condition for oxidation was 600 °C where nano-wires with the length of ~10 μm were fabricated.

There is some research in thermal spray technology where the aim is to incorporate metal oxide nano-second phase into metal thermal sprayed coatings to improve mechanical properties. For example, Limpichaipanit et al. [9] added Al₂O₃, ZrO₂ and TiO₂ nanoparticles into AlSi feedstock powder and thermal sprayed the powders with low velocity oxy-fuel (LVOF) technique. It was found that the coatings had higher hardness and sliding wear resistance. Kaewsai et al. [10] fabricated stainless steel/carbon nanotube composite coatings using chemical vapor deposition (CVD) technique and the coating had the increase of hardness ~60% and the wear rate of nearly 2 times lower than the pure stainless steel.

This work aims to investigate the effect of iron oxide on microstructure and mechanical properties of thermal sprayed stainless steel coatings produced by LVOF, which is the most primitive method of thermal spray. Iron oxide was obtained by direct addition deliberately or by thermal oxidation of iron (stainless steel feedstock powder). Physical properties (coating thickness, porosity and amount of iron oxide), microstructure (morphology of the coatings) and mechanical properties (hardness and sliding wear properties) were investigated to explain the relationship between microstructure and properties and to compare the properties of the coatings produced by different feedstock powders.

2. Experimental procedure

Stainless steel powder was of commercial grade (Sulzer Metco 42C, Switzerland) and the composition was 81.8% Fe, 16% Cr and 0.2% C. Iron oxide nanopowders had two forms—Fe₃O₄ (magnetite) and Fe₂O₃ (hematite)—and both had the particle size <50 nm and purity of 99.9% (Sigma Aldrich, USA). Stainless steel/iron oxide feedstock powders were prepared by direct addition of iron oxide or thermal oxidation from stainless steel starting powder. For direct addition, 2 wt.% of iron oxide was added to stainless steel powder and then mixed by wet ball milling in ethanol for 5 h. The slurry was dried in an oven, resulting in the composite powders for thermal spray. For the powder prepared by thermal oxidation, the stainless steel powder was washed using ethanol, dried in an oven and then placed in alumina crucible. To ensure that the powder had as much surface area as possible (to contact with air and undergo oxidation), the powder was spread to be very thin on the crucible. The powder was heated in air at 600 °C for 6 h with the heating and cooling rate of 5 °C/min. The feedstock powders were characterized by scanning electron microscopy (SEM-LV5910, JEOL, Japan) in conjunction with energy dispersive X-ray spectroscopy (EDX, Oxford Instruments, UK).

The stainless steel-based composites coatings were fabricated by low velocity oxy-fuel (LVOF) technique. Substrates made of mild stainless steel (dimensions of 1" × 2") were roughened by silicon carbide particle blast prior to flame spray. The substrates were cleaned with acetone to eliminate grease and dust particles. Flame spray was carried out using the parameters as shown in Table 1. SEM was used to see the microstructure of the coatings in cross sectional area. The thickness, porosity and amount of oxide of the coatings were determined using the SEM micrographs and ImageJ program. Determination of iron oxide compounds of the feedstock powders and coatings in terms of oxidation

state of iron was investigated by X-ray photoelectric spectroscopy (XPS, Kratos Analytical Ltd., UK).

In this work, hardness and sliding wear properties of the coatings were investigated. To prepare the samples for hardness measurement, the cross-sectioned coatings were polished to 1 μm finish. The indentation load was 300 g and the dwell time was 5 s. Vickers hardness values were obtained from 10 measurements. Pin-on-disk machine (Tribometer 2000, USA) was operated to perform sliding wear test (complied with ASTM G99-04 standard). The samples were polished to 1 μm finish and the test was carried out in air at 25 °C and the relative humidity (RH) of ~50–60%. The ball for sliding wear test was WC-Co. The sliding speed of 7.5 cm/s and applied load of 200 g was set for the wear test. Optical microscope was used to see wear tracks and the photographs of wear tracks were then used to determine wear track width and sliding wear rate of the coatings in terms of the volume loss (complied with ASTM G66-90). The sliding wear rate can be calculated by the equation:

$$volume = \frac{\pi(r_{track})(w_{track})^3}{6(r_{ball})}$$

where *volume* is the volume loss during the sliding wear in the units of mm³ (and wear rate in terms of volume loss per sliding distance is mm³/m), *r_{track}* is the maximum radius of wear track (3 mm), *w_{track}* is the wear track width in mm and *r_{ball}* is the radius of the ball (3.15 mm).

3. Result and discussion

The stainless powder had irregular shape with the particle size in the range of tens of microns (Fig. 1(a)). Fig. 1(b) is the micrograph of the stainless steel composite powder prepared by thermal oxidation of iron. Iron oxide nanoplatelets were found to be on the surface of stainless steel powder. The morphology was similar to the surface of oxidized stainless steel in air at elevated temperature reported by Higginson and Green [11] where the condition was 800 °C for 4 h. It was also similar to the "α-Fe₂O₃ nanoflakes" prepared by Wu et al. [12] via thermal oxidation at 500–600 °C for 5 h. It is generally accepted that nucleation and growth of metal oxide control the final nanostructure form, which is described in the literature and research work elsewhere [13–15]. The stainless steel composite powders prepared by direct addition of iron oxide nanoparticles are shown in Fig. 1(c) and (d). It can be seen that the nanoparticles were adhered to the stainless steel surface evenly, which was similar to the previous report [16,17].

The preliminary result of EDX (not shown here) revealed that there was oxygen (O) in the thermally oxidized powder. XPS results of the feedstock powders (as-received and thermally oxidized) are shown in Fig. 2. It can be seen that the powder prepared by thermal oxidation has the characteristic Fe 2p_{3/2} satellite at the binding energy of ~719 eV and Fe 2p_{1/2} satellite at the binding energy of ~732 eV. This confirms the presence of iron(III) oxide (α-Fe₂O₃) [18–20] resulting from oxidation whereas no such satellites are present in the as-received powder. The amount of iron oxide resulting from thermal oxidation can be calculated assuming that the oxide homogeneously covered on the surface of the initial feedstock powder (with the dimensions of 50 μm × 20 μm × 20 μm) as a thin layer of the thickness of 1 μm. The calculation based on the mentioned assumption gives the answer as 1.6 wt.% of iron oxide, which is slightly less than the amount of iron oxide deliberately added to the feedstock powder (2 wt.%).

Fig. 3 shows the typical microstructure of stainless steel thermal sprayed coatings. The coating was in the form of splats (lamellar structure resulting from the collision of semi-solid powder particles to the substrate or the splats previously deposited onto the surface) and unmelted particles, oxide layers between splats and pores can also be seen. The SEM micrograph in Fig. 3 is in backscatter mode so different contrast indicates different phases in the coating. The gray area is the oxide caused by oxidation at the surface during flame spraying [21,22] or melting of oxide in the coatings with oxide from addition or thermal

Table 1
Flame spray parameters.

Parameters		Units
C ₂ H ₂ (acetylene)	55	ft ³ /h
O ₂ (oxygen)	45	ft ³ /h
N ₂ (nitrogen)	3.5	ft ³ /h
Feed rate	75	g/min
Spraying distance	14	cm
Deposition time	180	s

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